EN

APPENDIX 4

001560

Certified Laboratories, Inc.

Degradation of Chlorite in Cow Manure

Final Report
March 15, 1999

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200 Express Street • Plainview, New York 11803 • www.800certlab.com

DEGRADATION OF CHLORITE IN COW MANURE: FINAL REPORT

I. OBJECTIVE

To estimate the rate of degradation of the chlorite ion in cow manure, under simulation of conditions applicable to use of 4XLA Teat Dip.

II. BACKGROUND

The study is being undertaken at the request of Dr. Robert Kross, to satisfy regulatory requirements in the Netherlands. Some parameters of the study are therefore specified by the test protocols in use under the regulatory regime. In particular, the regulations specify that this study be done at a temperature of 10°C.

III. TECHNICAL ISSUES

The total rate of degradation of chlorite ion is determined by the sum of the rate of reduction of chlorite by materials in the manure and that of any auto-degradation of the chlorite. In a matrix with a high organic load, reduction is likely to be dominant. Under these conditions we believe that the kinetics are likely to be simple as long as the reducing capacity of the matrix is large compared to the amount of analyte added to the matrix. In effect the process will then behave as if it were zero order in terms of constituents of the matrix.

Further, the spiking level chosen for the experiment should be at least as high as any levels likely in the real-life application.

The teat dip contains about 2400 ppm of chlorite ion. If we assume that any spillage of the teat dip is likely to be diluted by a factor of at least 10 by dispersion in cow manure we expect an initial concentration of chlorite ion equal to no more than 240 ppm in the manure. This would appear to be a reasonable a priori assumption.

Prior experience with chlorite determinations leads us to expect detection limits in the 5-20ppm range in a matrix with high levels of co-extractibles. We believe, therefore, that a spiking level of 240 ppm chlorite will meet both of the above requirements. The concentration is high enough to represent a reasonable simulation of the actual conditions and makes for relatively straightforward quantitation, yet low enough that it is unlikely to affect the substrates in the matrix to any great extent.

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DEGRADATION OF CHLORITE IN COW MANURE: FINAL REPORT

This study will attempt to determine whether, under these conditions, the concentration of chlorite ion in cow manure falls below detectable levels within a month.

We believe that the reaction rate is likely to be quite high and that the chlorite level is likely to fall to half, under these conditions, in minutes or hours. It should be sufficient then to monitor chlorite levels at intervals of 10 or 20 minutes.

If little or no change is seen over a few such periods, it remains easy to increase the intervals.

If it turns out that the rate is much lower, it may become necessary to extend the experiment into a second day.

If it turns out that the reaction rate is sufficiently low that multiple days are required for the level to fall to half the initial concentration, it will become necessary to redesign the experiment, with considerable change in logistics.

Finally, some modification of the matrix may be desirable to ease handling and sampling, in particular to ensure that added analyte is properly distributed through the matrix. This will require some dilution of the matrix with water. It is reasonable to suppose that as long as this dilution is not excessive, the experiment will not be compromised. Further, the worst-case is that reaction rates will be lower than in the undiluted matrix. If that happens, the experiment will produce an under-estimate of the rate of degradation, i.e. we will have an overestimate of the time required for the chlorite ion level to fall to half under the conditions. As long as this overestimate is itself shorter than one month, we will have achieved the purposes of this study.

Discussions with Dr. Kross lead us to expect that a 1:1 aqueous dilution is likely be satisfactory. This is, of course, subject to adjustment based on experience.

IV. EQUIPMENT/MATERIALS

1. HPLC system, isocratic, with autosampler, UV detector at 214nM and Waters Associates IC-PAK Anion HC column, 4.6mm id X 150mm, guard column, borate-gluconate mobile phase specified by Waters Associates.
2. Sodium tetraborate, gluconic acid and acetonitrile (for mobile phase)

3. Sodium chlorite stock solution (supplied by Dr. Kross)
4. Syringe filters, syringes and other consumables as needed
5. Miscellaneous laboratory glassware as needed
6. Circulating water bath or other suitable equipment for maintenance of temperature at 10° C

V. METHODOLOGY

1. Obtain fresh cow manure. Store under refrigeration until use.
2. Prepare a solution containing 240 ppm chlorite ion, at a pH of 10. Call this solution A. Bring to 10° C.
3. Prepare a solution containing 240 ppm chlorite ion in deionized water. Call this solution B. Bring to 10° C.
4. Bring the manure to 10° C.
5. To a known weight of manure at 10° C, add a quantity of solution A. such that there is 240 ppm chlorite ion on original basis, i.e. 1 ml of solution per gram of manure. Mix well. Dilute with known amount of water to ensure appropriate mechanical handling.
6. Immediately remove an aliquot and filter for hplc.
7. Determine level of chlorite in the extract, expressed as ppm relative to original weight of manure. This provides a time zero level.
8. To additional weighed portions of manure at 10° C add deionized water, also at 10° C. Mix well and maintain at 10° C. Use these as blanks. Ensure that the manure to water ratio is the same as in the rest of this study.
9. To additional weighed portions of manure at 10° C add solution B, also at 10°C, so as to have 240ppm chlorite on original manure basis. Mix well and maintain at 10° C. Dilute with water as for the time zero experiment.
10. Immediately remove aliquots from blank and sample mixtures and determine the level of chlorite. If this level in the sample extract is lower than the time 0 level as determined above, make careful judgements as to any need to stabilize analytical aliquots by adjusting pH.
11. At intervals thereafter, take aliquots; adjust pH if appropriate based on information derived from step 9. Determine levels of chlorite in these aliquots.
12. Examine the kinetics to obtain an estimate of the rate of degradation.

The experiment will be continued for up to 48 hours, the exact period to be determined based on evaluation of the data as it is collected.

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VI. FINAL REPORT

The final report will provide complete documentation of the conditions of the experiment. Results will be presented graphically, as a plot of the chlorite concentration against time.

The blank data will be used to determine if any baseline problems develop as the experiment proceeds.

MODIFICATIONS FROM ORIGINAL PROTOCOL

Actual concentration of chlorite used in the study:

The chlorite solutions used in this study were prepared by dilution of a 30.7% solution of sodium chlorite provided by Dr. Robert Kross. Convenient serial dilutions produced solutions with 229 ppm chlorite ion. This was deemed sufficiently close to the concentration proposed in the protocol to meet all requirements of the study.

Solutions at this concentration were prepared in deionized water and in dilute sodium hydroxide (0.01 N).

Actual dilution relative to manure:

Preliminary experiments showed that in order to obtain appropriate analytical aliquots of the sample extracts a net dilution of 4:1 relative to original manure was needed.

Accordingly portions of manure were prepared as follows:

Blanks:
A known weight of manure was diluted with 3 ml of water per gram of manure, for a net dilution of 4. Multiple blanks were set up. A given "blank tube" was discarded when it was no longer convenient to obtain analytical aliquots from it.

Time zero experiment:
To a known weight of manure 1 ml of alkaline chlorite solution (229 ppm chlorite ion in 0.01 N sodium hydroxide) per gram of manure was added to produce a mixture with a chlorite level equal to 229 ppm on a manure basis. This mixture was further diluted with 2 ml of water per gram of manure, to produce a net dilution by 3 ml of aqueous media per gram of manure, for a net dilution of 4 times.

Degradation study:
To a known weight of manure 1 ml of chlorite solution (229 ppm chlorite ion in deionized water) per gram of manure was added to produce a mixture with a chlorite level equal to 229 ppm on a manure basis. This mixture was further diluted with 2 ml of water per gram of manure, to produce a net dilution by 3 ml of aqueous media per gram of manure, for a net dilution of 4 times.

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Multiple sub-samples were set up for the degradation study. These sub-samples were placed at 10° C. An analytical aliquot was removed and analyzed after a

specified period of time. Each sub-sample was discarded after sampling for liquid chromatography. Exact weights of manure were recorded in each instance.

Chlorite solutions and water were added using 10ml graduated pipettes.

MATERIALS AND METHODS

Materials and methods are documented in this section only as needed for clarification of detail relative to the protocol.

Fresh manure was collected at a farm and maintained at refrigerator temperatures.

Preparation and handling of test mixtures:

The manure and reagents were brought to 10° C in a water bath prior to mixing. The mixtures were maintained at 10° C for the appropriate period of time, then centrifuged for 5 min and an aliquot was transferred to a 3 ml polypropylene syringe fitted with a syringe filter with a PTFE membrane with 0.45µ pore size. The filtrate was collected for HPLC analysis.

50 ml polypropylene centrifuge tubes with screw-caps were used for samples, blanks and the time zero experiment.

A single portion was used for the time zero experiment. The solution of chlorite in 0.01 N sodium hydroxide did not raise the pH meaningfully, so a few drops of 25% sodium hydroxide solution were added to raise the pH to just above 10.

One sub-sample (and therefore one tube) was prepared for each interval in the degradation experiments. Individual tubes were discarded after aliquots were taken for HPLC analysis.

Blanks were reused. After portions were removed for HPLC analysis the manure was re-suspended in the medium by shaking the tube thoroughly and the tube was maintained again at 10° C.

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Chromatographic conditions:

Mobile phase for HPLC:

Sodium gluconate (16 g), boric acid (18 g) and sodium tetraborate decahydrate (25 g) were dissolved in about 500 ml deionized water. Glycerin (250 ml) was added and the mixture was made up to 1 L with deionized water. This concentrate was kept in the refrigerator and used as needed for production of mobile phase.

The actual mobile phase was made by taking 20 ml of the above concentrate and 120 ml of acetonitrile and making up to 1 L with deionized water.

A flow rate of 2 ml/min provided retention time of about 4 min for chlorite.

A baseline dip was seen shortly before the chlorite peak. This dip was essentially constant over the duration of the experiment. The size of the chlorite peak was estimated as the height of the peak above the nominal baseline.

Using this height as the estimate of response, chlorite ion concentration was calculated as final concentration in the solution. The time zero experiment represents 229 µg chlorite ion per gram of manure. The actual concentration in solution is, of course, lower, because of the dilution of the sample.

The analytical standards were of 57.2 µg/ml concentration, expressed as chlorite ion.

Alkaline solutions provided slightly elevated response relative to solutions made in deionized water. Consequently the time zero experiment was analyzed against a standard solution prepared in 0.01 N sodium hydroxide.

The degradation samples were analyzed against a standard prepared with deionized water and maintained in the refrigerator to minimize auto-degradation of chlorite. This standard showed quite constant response at the hplc over the period of the experiment.

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DEGRADATION OF CHLORITE IN COW MANURE: FINAL REPORT

OBSERVATIONS

Final concentrations of chlorite ion in the test mixtures are shown below. Exposure time includes time spent in the centrifuge. Concentration on a manure basis is estimated by normalizing the time zero value to 229 ppm to account for the effective dilution of the experiment.

Exposure time (hours)	Chlorite concentration in solution (ppm)	Chlorite concentration on manure basis (ppm)
0	56	229
0.33	53	217
0.75	52	213
1.25	11	45
4	11	45
5	12	49
15	11	45
24	5	20
48	5	20

Blanks increased slightly over the course of the experiment. At the beginning they showed a peak height equivalent to about 1 ppm chlorite in solution or about 4 ppm on a manure basis. At the end of 48 hours the blanks were at about 3 ppm chlorite in the final solution, or about 12 ppm on a manure basis

Treatment of the 48 hour extract with a few crystals of sodium thiosulfate does not cause any immediate change in the size of the chlorite peak. It therefore seems unlikely that the residual peak at that stage is actually chlorite. It may represent the background level of interference from some species in the matrix. It is, however, not clear why the peak is somewhat larger than the blanks seen during this experiment.

Attached are copies of the relevant chromatograms for blanks, the time zero experiment and the extracts for the degradation experiment. It should be noted that the decline in chlorite concentration is quite rapid and that in less than two days under the experimental conditions the chlorite seems to have disappeared.

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DEGRADATION OF CHLORITE IN COW MANURE: FINAL REPORT

CONCLUSIONS

- The rate of loss of chlorite ion in manure is quite high.
- The chlorite is essentially completely consumed in less than 24 hours. The duration of this experiment appears to provide an upper limit on the period of persistence of chlorite.
- At a level of 229 ppm on manure basis the reducing capacity of manure is not exceeded.

[redacted]

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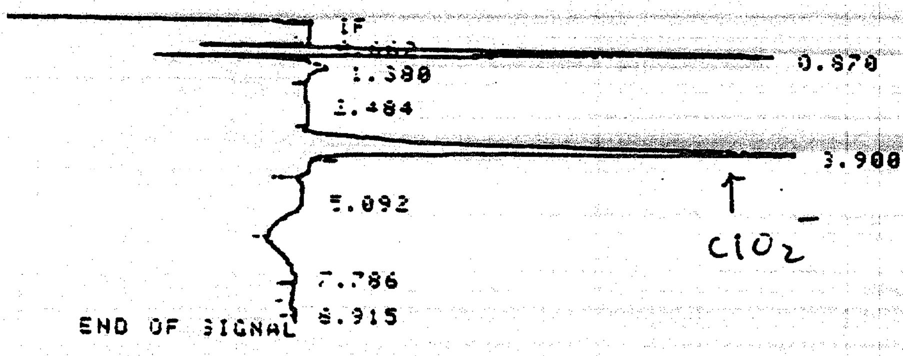
[redacted]

. 55	. .	105566	.357	3580		.000
5.915	.	19274	.303	1059		.000

TAL AREA=7845370
FACTOR=1.0000E+00

LIST: ATT 2^ =
ATT 2^ 5

689 FEB 9, 1999 11:07:00
ART



std ClO_2^- in alkali
57 ppm ClO_2^-

sing signal file A:SIGNAL .BNA

689 FEB 9, 1999 11:07:00

PLE NAME: MANURE
CPAK TS2101A

NTIFIER : W/ALKALI 10-
NAL FILE: A:SIGNAL.BNA

ORITE

I-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	PPM	NAME
.662	P	406677	.261	25962		.000	
.870	PP	1477348	.151	135989		.000	
.380	PV	1645322	.705	38932		.000	
.484	V	1572075	1.194	21938		.000	
.900	VB	2332357	.344	112983	1R	49.484	CHLORITE
.092	BP	135600	1.350	2292		.000	
.786	PV	105566	.357	3580		.000	
.915	V	19274	.303	1059		.000	

L AREA=7845370
FACTOR=1.0000E+00

[redacted]

001571 [redacted]

[redacted]

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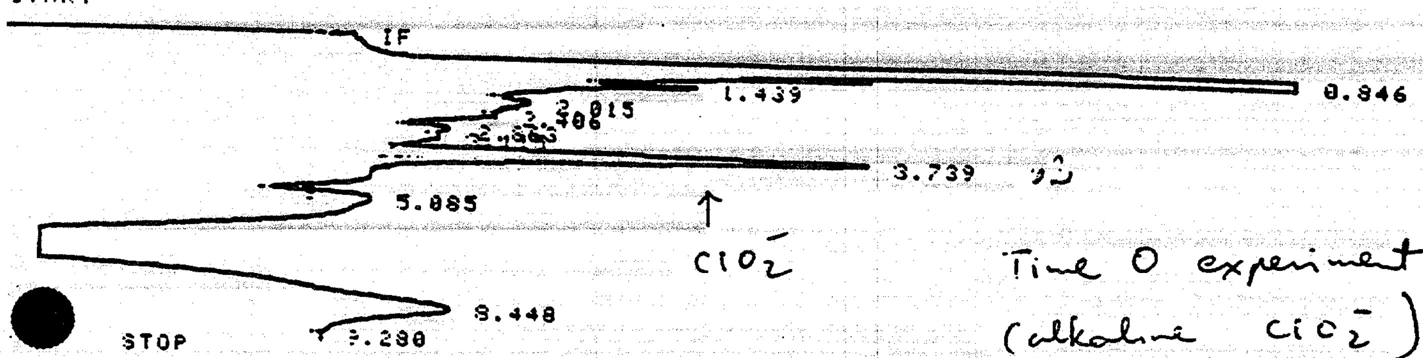

losing signal file M:SIGNAL .BNC

UN# 690   FEB 9, 1999 12:46:50

AMPLE NAME: MANURE
MICPAK TS2101A

DENTIFIER : T0
IGNAL FILE: M:SIGNAL.BNC

HLORITE

CALIB PEAKS FOUND
IGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
.846	1306502	BB	.274	80.54639
1.439	23188	BP	.150	1.50621
2.015	10893	[illegible]	.189	.67408
2.406	12381	[illegible]	.146	.76619
2.563	10445	[illegible]	.221	.64636
[illegible]15	[illegible]	[illegible]	.196	[illegible]
[illegible]	106741	[illegible]	[illegible]	[illegible]
[illegible]085	36112	[illegible]	[illegible]	[illegible]
8.448	[illegible]	[illegible]	[illegible]	4.[illegible]
9.280	19902	[illegible]	[illegible]	[illegible]

AL HEIGHT=[illegible]

001572

[redacted]

[redacted]

STD WH20-10-25

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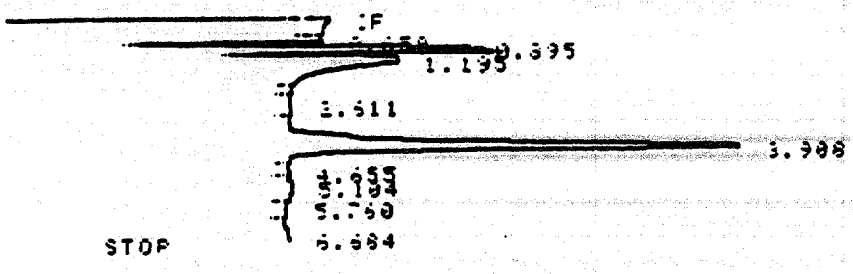

standard ClO_2^- in water

57 ppm ClO_2^-

Closing signal file M:SIGNAL .BNC

RUN# 692 FEB 9, 1999 13:08:43

SAMPLE NAME: MANURE
DIONEX ICPAK TS2101M

IDENTIFIER : STD WH20-10-
SIGNAL FILE: M:SIGNAL.BNC

WRITE

STD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	%	NAME
.659	BP	198606	.137	24138		.000	
.895	PV	910956	.216	70269		.000	
1.195	VV	2122387	.664	53302		.000	
2.611	VV	373116	.644	22708		.000	
3.908	VV	2959240	.431	114539	1R	155.563	CHLORITE
4.655	VV	415080	.591	11715		.000	
5.104	VV	130439	.638	3946		.000	
5.760	VV	156398	.491	5328		.000	
6.684	I VP	76057	1.197	1059		.000	

TOTAL AREA=4809378

AL FACTOR=1.0000E+00

001573

[redacted]

41

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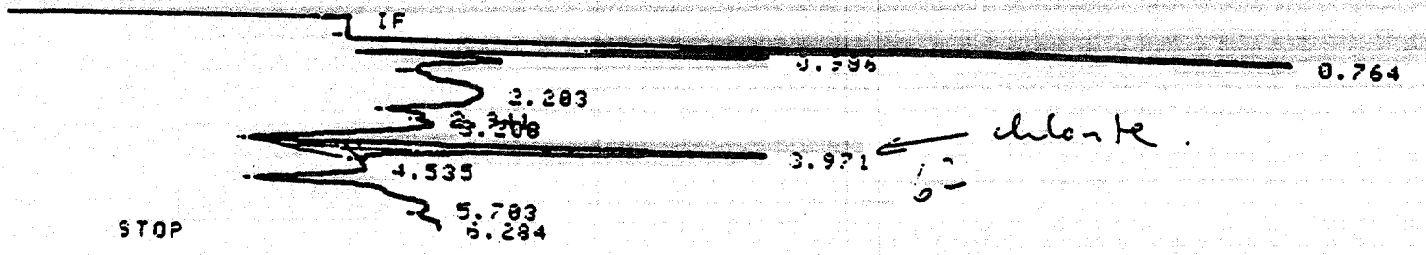

chlorate.

≤3

Degradation experiment
20 minutes exposure

losing signal file M:SIGNAL .BNC

UN# 693 FEB 9, 1999 13:23:30

AMPLE NAME: MANURE
/ICPAK T32101A

DENTIFIER : 41
IGNAL FILE: M:SIGNAL.BNC

ITE

I CALIB PEAKS FOUND
IGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
.764	418172	ISPB	.114	58.92869
.986	50409	ITBB	.002	7.10362
2.203	22623	BV	.856	3.18803
2.941	23591	VV	.247	3.32444
3.208	30299	VP	.444	4.26972
3.971	113065	PV	.268	15.93309
4.535	23765	VP	.451	3.34896
5.703	19344	PV	.858	2.72665
6.284	8351	VB	.568	1.17682

TAL HEIGHT= 709614
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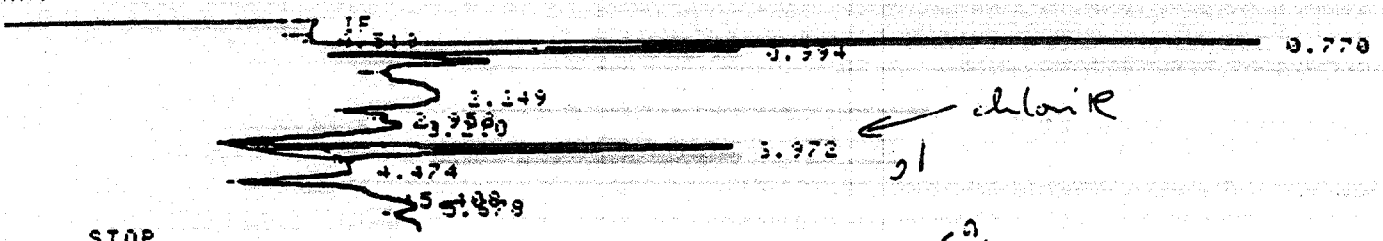
001574

[redacted]

RUN # 694 FEB 3, 1999 14:20:07

APT



chloride

STOP

Dejadation experiment
45 minutes total exposure

osing signal file M:SIGNAL .BNC

N# 694 FEB 3, 1999 14:20:07

MPLE NAME: MANURE
ICPAK T82101A

ENTIFIER : 43
GNAL FILE: M:SIGNAL.BNC

TE

CALIB PEAKS FOUND
IGHT%

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
.519	1016	BH	.094	.15092
.770	440754	ISHB	.114	65.46954
3.249	21823	BV	.806	3.24158
3.958	22336	VV	.229	3.31779
4.210	29533	VP	.455	4.38683
3.972	110093	PV	.364	16.35319
4.474	25153	VP	.462	3.73622
5.678	22513	PV	.300	3.34393

TAL HEIGHT= 673330
L FACTOR=1.0000E+00

001575

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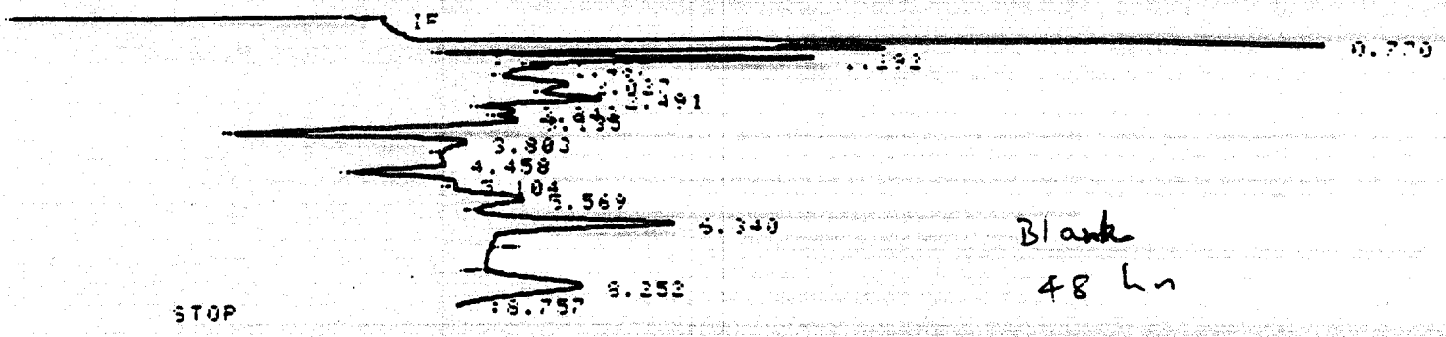

Blank
48 hr

Closing signal file M:SIGNAL .BNC

RUN# 713 FEB 11, 1999 10:34:04

SAMPLE NAME: MANURE
G/ICPAK T32101A

IDENTIFIER : BLANK 0
SIGNAL FILE: M:SIGNAL.BNC

CHLORITE

ESTD-AREA

PT	TYPE	AREA	WIDTH	HEIGHT	CAL#	%	NAME
.770	SBB	3603160	.126	477892		.000	
1.292	BV	1265007	.207	101842		.000	
1.467	VV	811328	.304	44430		.000	
2.027	VV	1459998	.441	55203		.000	
2.491	VV	1550256	.382	67670		.000	
2.843	VV	841211	.369	52178		.000	
3.135	VP	1281600	.379	56385		.000	
3.803	PV	1167039	.423	46017	IP	51.350	CHLORITE
4.458	VV	1053978	.559	31400		.000	
5.104	VV	508161	.353	24002		.000	
5.569	VV	1067844	.556	31023		.000	
6.340	VB	1441655	.447	53739		.000	
8.252	BP	743609	.517	33964		.000	

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MUL FACTOR=1.0000E+00

001576

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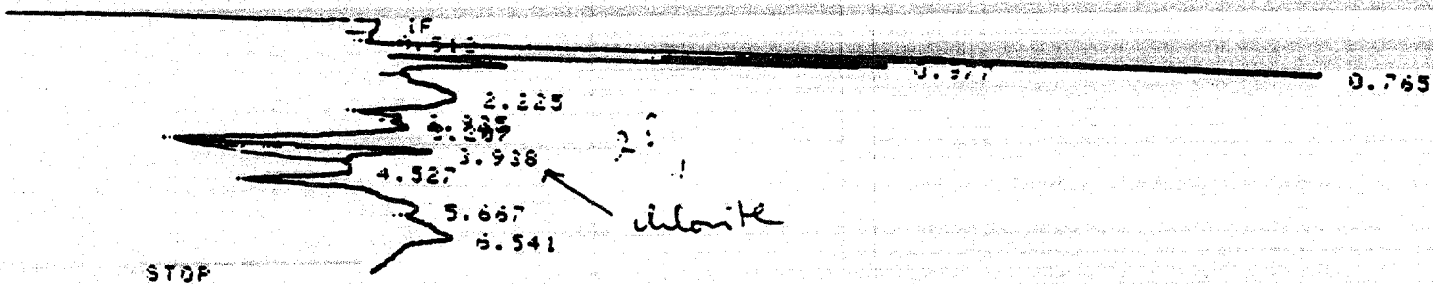

Degradation experiment
85 minute total exposure

Closing signal file M:SIGNAL .BNC

RUN# 696 FEB 9, 1999 15:47:59

SAMPLE NAME: MANURE
D:ICPAK TS210.M

IDENTIFIER : #4
SIGNAL FILE: M:SIGNAL.BNC

CHLORITE

PEAK-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	%	NAME
.512	PP	2758	.087	533		.000	
.765	ISPB	3336965	.127	437771		.000	
2.225	BV	1239735	.822	25133		.000	
2.925	VV	453528	.252	29983		.000	
3.207	VP	1085523	.480	37701		.000	
3.938	PV	1110929	.347	53417	IR	58.400	CHLORITE
4.527	VV	898282	.501	29901		.000	
5.667	VV	1541824	.789	32567		.000	
6.541	VP	2012057	1.089	30794		.000	

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AL FACTOR=1.0000E+00

001577

-5

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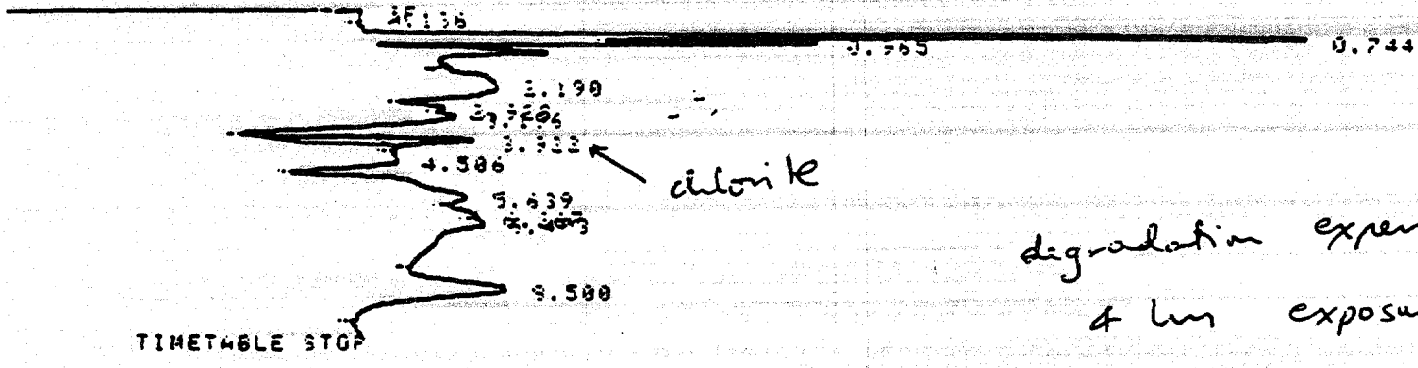

chlorite

degradation experiment
4 hrs exposure

Closing signal file M:SIGNAL .SNC

RUN# 699 FEB 9, 1999 19:22:05

SAMPLE NAME: MANURE
G:\ICPAK TS2101M

IDENTIFIER : -5
SIGNAL FILE: M:SIGNAL.SNC

CHLORITE

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	%	NAME
.136	PP	1893	.134	361		.000	
.744	ISPB	1761854	.107	432271		.000	
.965	ITBB	133856	.038	58471		.000	
2.190	BV	961145	.830	19294		.000	
3.928	VV	371575	.240	25846		.000	
3.196	VP	586661	.476	34641		.000	
3.922	PV	599696	.335	49797	1P	52.563	CHLORITE
4.506	VV	1005522	.541	30973		.000	
5.639	VV	1330782	.751	41209		.000	
6.367	VV	1011516	.415	40723		.000	
8.473	V	1565301	1.040	41091		.000	
8.500	P	1559475	.529	47308		.000	

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DIL FACTOR=1.0000E+00

001578

[redacted]

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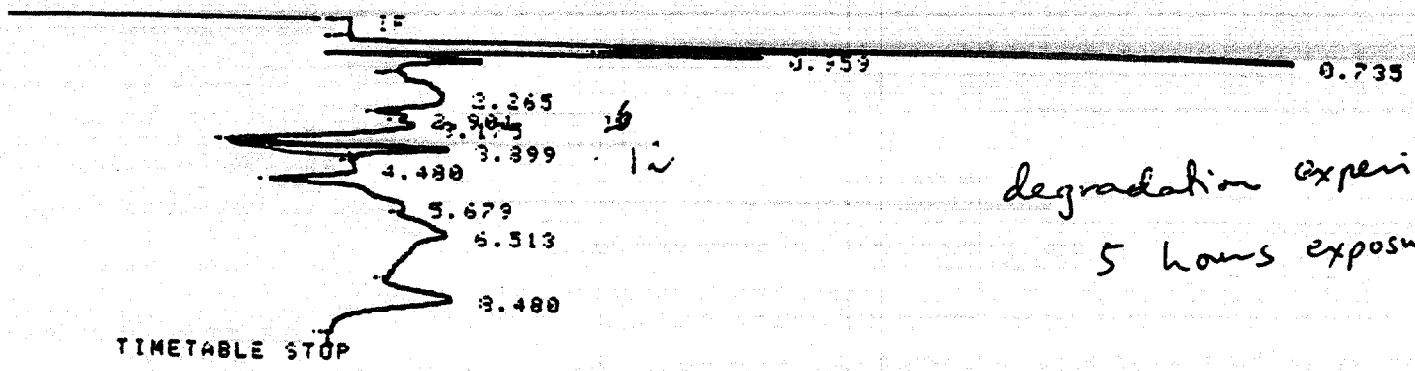


TIMETABLE STOP

degradation experiment
5 hours exposure

losing signal file H:SIGNAL .BNC

UN# 700 FEB 9, 1999 19:39:28

AMPLE NAME: MANURE
ICPAK TS2101A

.FIER : #6
AL FILE: H:SIGNAL.BNC

LORITE

TD-AREA

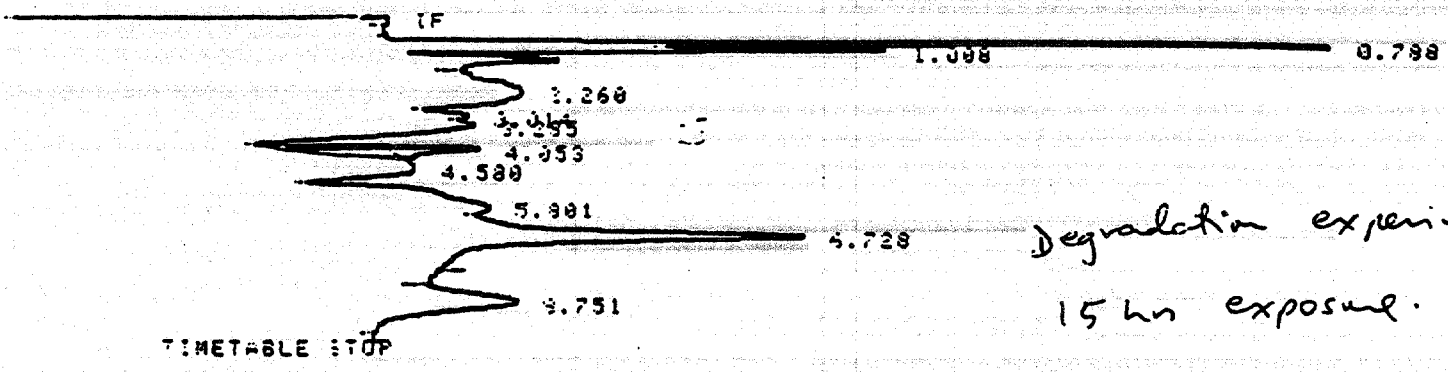
RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	%	NAME
.735	ISBB	2467173	.134	307673		.000	
3.265	BV	989738	.803	20536		.000	
2.901	VV	379933	.263	24044		.000	
3.175	VP	527701	.452	30497		.000	
3.899	PV	916164	.328	46507	:P	48.162	CHLORITE
4.480	VV	755968	.514	24489		.000	
5.679	VV	1450415	.787	30729		.000	
6.513	VV	3389166	1.933	37102		.000	
8.480	VV	1579742	.640	31147		.000	

TAL AREA=1.[illegible]E+07
FACTOR=1.0000E+00

001579

*ID B2

* RUN # 705 FEB 10, 1999 12:00:54
START



Degradation experiment
15 hr exposure.

TIMETABLE STOP

Closing signal file M:SIGNAL .BNC

RUN# 705 FEB 10, 1999 12:00:54

SAMPLE NAME: MANURE
S/ICPAK T821018

IDENTIFIER : B2
SIGNAL FILE: M:SIGNAL.BNC

CHLORITE

ESTD-AREA

PT	TYPE	AREA	WIDTH	HEIGHT	CAL#	%	NAME
.788	ISPB	1165501	.122	434177		.000	
2.260	BV	1330547	.682	35143		.000	
3.014	VV	450290	.261	28767		.000	
3.265	VP	1057346	.493	35778		.000	
4.053	PV	915470	.328	46554	IR	48.125	CHLORITE
4.580	VP	381591	.533	17567		.000	
5.801	PV	1451887	.754	32097		.000	
6.728	VB	1161616	.568	31851		.000	
6.751	BP	722216	.715	23382		.000	

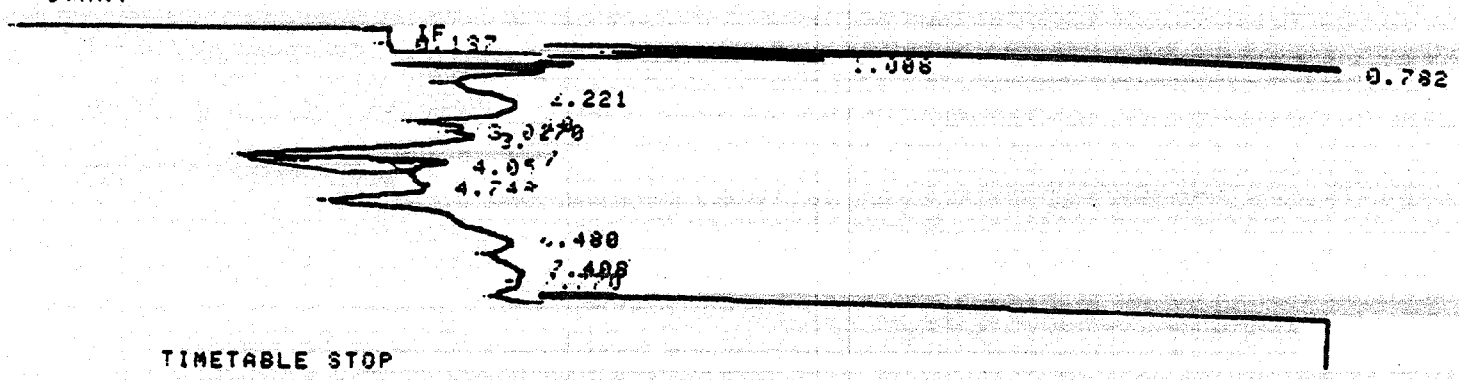
TOTAL AREA=1.1116E+07
MUL FACTOR=1.0000E+00

001580

*ID B3

*ISAD
B3

* RUN # 709 FEB 10, 199? 20:19:41
START



TIMETABLE STOP

Closing signal file M:SIGNAL .BNC

Degradation experiment
24 hrs exposure

RUN# 709 FEB 10, 1999 20:19:41

SAMPLE NAME: MANURE
G/ICPAK T021018

IDENTIFIER : B3
SIGNAL FILE: M:SIGNAL.BNC

CHLORITE

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	%	NAME
.137	BV	2065	.	263		.000	
.782	ISHB	2746704	.	413377		.000	
1.008	ITBB	30949	.	52567		.000	
2.221	BV	1290167	.	24119		.000	
3.027	VV	427539	.	13789		.000	
3.270	VP	1139909	.	36393		.000	
4.057	PV	847073	.	41137	1R	44.529	CHLORITE
4.743	VV	1470696	.	72480		.000	
6.480	VV	2961808	.	58203		.000	
7.408	VV	1433846	.	33504		.000	
7.770	VV	745645	.	30308		.000	

001581

TOTAL AREA=1.1125E+07
TOTAL F=ITOP=1.0000E+00

[redacted]

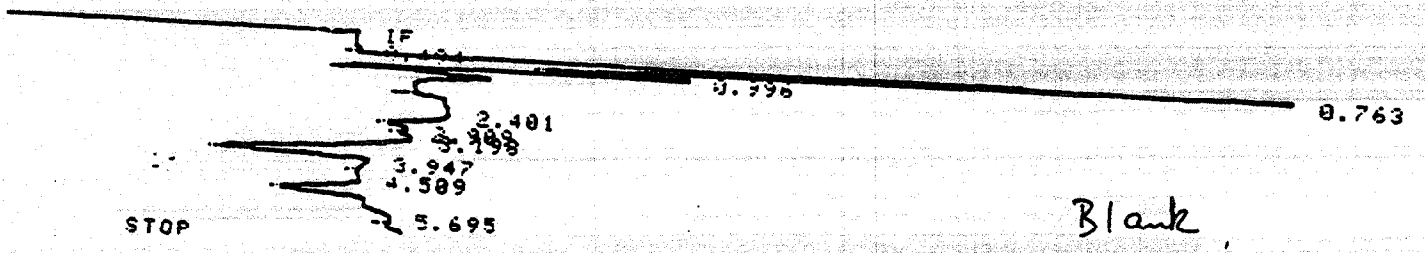
OP # 4

REPORT OPTIONS

Suppress local report [Y/N*]:
HEIGHT% report [Y*/N]:
Replace report title [Y/N*]:
Replace amount label [Y/N*]: Y
Amount label: %
Report uncalibrated peaks [Y*/N]:
Extended report [Y*/N]:

*ID BLANK

* RUN # 691 FEB 3, 1999 13:01:22
START



Blank
start of experiment

ns signal file M:SIGNAL .SNC

UN# 691 FEB 3, 1999 13:01:22

AMPLE NAME: MANURE
ICPAK TS2101A

DENTIFIER : BLANK
IGNAL FILE: M:SIGNAL.SNC

ILORITE

CHLIB PEAKS FOUND
IGHT%:

RT	HEIGHT	TYPE	WIDTH	HEIGHT%
.494	1058	BM	.200	.36279
.763	270790	BMB	.130	97.26032
2.401	13354	BV	.094	4.80725
2.909	15292	BV	.095	5.51317
3.198	20575	B	.482	7.42052
3.947	19527	BV	.393	7.03498
4.509	15140	VB	.539	5.44958
5.695	3864	BV	.045	1.35330

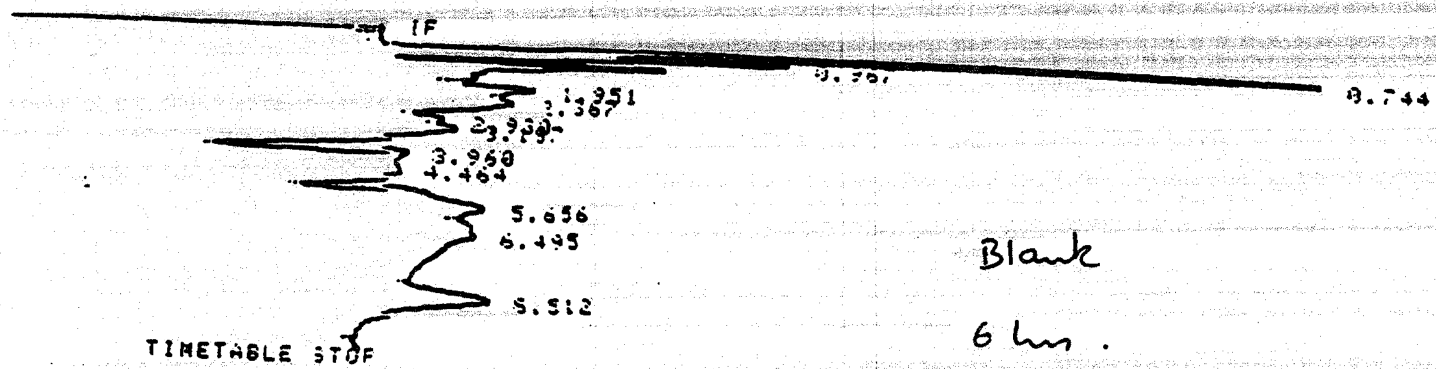
EIGHT= 402600
TOP=1.0000E-00

001582

ID BLANK.6HRS

*ID
BLANK.6HRS

* RUN # 698 FEB 9, 1999 19:08:41
START



Blank

6 hrs.

Closing signal file M:SIGNAL .BNC

RUN# 698 FEB 9, 1999 19:08:41

SAMPLE NAME: MANURE
CPAK T821014

IDENTIFIER : BLANK.6HRS
SIGNAL FILE: M:SIGNAL.BNC

CHLORITE

ESTD-AREA

RT	TYPE	AREA	WIDTH	HEIGHT	CAL#	%	NAME
.744	ISFB	16040[illegible]	.103	42147S		.000	
.967	ITBB	24354[illegible]	.083	48966		.000	
1.951	BV	35444	.273	21885		.000	
2.367	VV	55404	.539	17550		.000	
2.930	VV	41112[illegible]	.240	13555		.000	
3.167	VP	167[illegible]	.5[illegible]	1310[illegible]		.000	
3.960	PV	90256[illegible]	.360	4105[illegible]	1R	[illegible].447	CHLORITE
4.464	VV	130465	.559	36903		.000	
5.656	VV	253120	.561	44015		.000	
6.495	VV	194460	1.5[illegible]	42590		.000	
5.512	VV	173025	.505	15803		.000	

TOTAL AREA=1.5048E+07
MUL FACTOR=1.0000E+00

001583 [redacted]

[redacted]

[redacted]